UDC 666.1.056:539.217

DETERMINATION OF THE WET STRENGTH OF OXIDE COATINGS ON GLASS

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Translated from Steklo i Keramika, No. 10, pp. 6 – 7, October, 1998.

Variations in the optical thickness of oxide coatings on glass in treatment with benzoic acid melts are investigated by the ellipsometric method. The possibility of rapid estimation of their wet strength is shown.

The constantly growing need for optical oxide coatings calls for implementation of efficient and fast methods of control of their wet strength. The wet strength of optical coatings is tested according to OST 3-1901–95 methodology. Holding of glass articles with deposited coatings for 10 days in an environment with a relative humidity of 95 – 98% (without moisture condensation) at the temperature of 40°C is accompanied by visual monitoring of surface flaws.

The chemical resistance of the pieces with coatings referred to mechanical strength groups 0, I, and II is tested by immersion of the piece in a decinormal aqueous solution of CH_3COOH with pH = 2.7 (GOST 61–75) for 1 min. Depending on the loading, the coated pieces are tested for alkali resistance by treatment in aqueous solutions of NaOH with pH = 12.7. The strength tests include as well exposure to temperatures up to 450°C for 2 h at a heating rate of 30°C/h. According to this method, the mechanical strength is determined by the number of abrasion cycles before a rubber tip with cambric produces a through circular scratch. The whole set of strength tests is very cumbersome and is performed only on 3% of the coated pieces.

The purpose of the present study was to improve the process of determination of the wet strength of oxide coatings taking into account the available data from [1, 2], without damaging the glass articles.

Coatings based on Al, Sc, Ti, and Zr oxides, and $ZrO_2 - Sc_2O_3$ and $ZrO_2 - Y_2O_3$ systems were selected for testing. The coatings were produced at different speeds of electron-beam evaporation of the pelleted initial mixtures. The articles (prisms made of K-8 glass) with deposited coatings 0.2 – 0.8 μm thick were treated in C_6H_5COOH melts at the temperature of 150 – 180°C for 5 – 20 min. The ellipsometric analysis of the coating material before and after treatment was performed on a LEF-3M ellipsometer

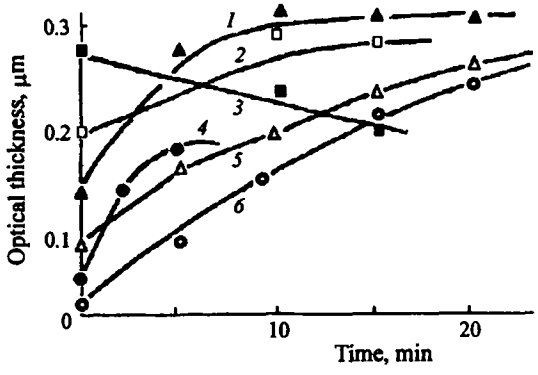

Fig. 1. Variations in the optical thickness of coatings based on SiO_2 (*1*), Sc_2O_5 (*2*), ZrO_2 (*3*, *4*), and Al_2O_3 (*5*, *6*) versus time of treatment in C_6H_5COOH melts at the temperature of 150°C and deposition rate. The deposition rate is 200 (*2*, *6*), 300 (*4*, *5*), 400 (*1*), and 600 (*3*) Å/min.

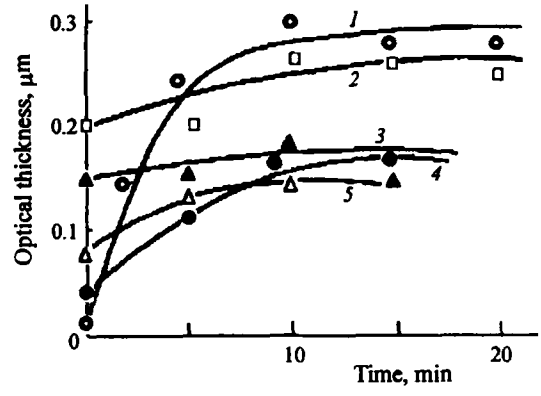

Fig. 2. Variations in the optical thickness of coatings based on ZrO_2 with addition of Sc_2O_3 versus time of treatment in C_6H_5COOH melts at the temperature of 150°C. The molar content of Sc_2O_3 is 5 (*1*), 80 (*2*), 50 (*3*), 40 (*4*), and 8% (*5*).

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0361-7610/98/0910-0303$20.00 © 1999 Kluwer Academic/Plenum Publishers

($\lambda = 0.63$ μm) using the model of isotropic coating on an isotropic article. The quality of the coating was characterized by the relative density related in the following way to the refractive index [3]:

$$p = \frac{n_1^2 - 1}{n_1^2 + 2} \frac{n_2^2 + 2}{n_2^2 - 1},$$

where n_1 and n_2 are the refractive indexes of the article coating and the glass.

The coating density was registered on the basis of the variations in optical thickness. It was found from the dependences in Fig. 1 and visual surface control that etching traces were identified only in the coatings whose optical thickness decreased with an increase in the time of treatment. With a low density of the coating material and existence of inclusions, the formation of hydroxyl groups is accompanied by their condensation around cations and hydration of the oxide with the latter passing into the melt. This results in etching of the coatings that is visually observed. The quality of the coating can be assessed by the extent of the corrosion.

With an increase in the optical thickness, the resistance of the coatings to destruction increases, which is related to an increase in the concentration of polarized state protons and the absence of hydration. The invariability of the optical parameters of the coatings after melt treatment can likewise be related to an increase in their chemical resistance. With a different ratio of components in coatings based on oxide systems, such treatment is accompanied by an increase in the refractive index or destruction of the surface. In the coatings based on the $ZrO_2 - Sc_2O_3$ system with a molar content of Sc_2O_3 from 8 to 20%, low chemical resistance corresponded to a decrease in the optical thickness (Fig. 2). Addition of up to 13% Y_2O_3 to ZrO_2 causes an increase in the resistance of the coatings to hydration and destruction.

Thus, an efficient method for assessing of the moisture resistance of optical oxide coatings consists in treatment of optical glass articles in anhydrous medium, i.e., benzoic acid melt, at a temperature from 150 to 180°C for a period of 5 – 20 min with subsequent control of the optical thickness using the ellipsometric method.

Partial dissociation of water molecules on holding the coatings in humid atmosphere results in the formation of H^+ and OH^- ions. The latter react with the surface and form a layer preventing penetration of protons into the depth of the coating. In using melts of benzoic acid, which is a good source of protons (without OH^- ions), the effect of restriction of proton incorporation in the tested coating is absent.

The treatment of optical coatings on glass articles in anhydrous melts of benzoic acid results in corrosion of oxides in areas of low density and weak adhesion to the coated surface. In the absence of corrosion, saturation of the oxides with protons is accompanied by an increase in the optical thickness of the coatings.

The developed method for wet strength assessment does not produce negative phenomena in dense coatings with high adhesion and can be extended to other materials.

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